

Section XV: Identification of Hallucinogenic Mushrooms

I. Introduction:

Peyote Buttons are first given a visual test to check for their characteristic. The peyote buttons are the top of the cactus, only about two inches in diameter and ½ inch tall, they are rounded and grayish-green in color, there are no spines on the cactus but it does have short tuft of hair growing on top.. Then the peyote sample are solvent extracted using Ethyl Ether. This extract is then run on the GC/MS. If the peyote sample is positive the active ingredient Mescaline will be detected on the GC/MS.



II. Reagents:

- A.) 3 N Acetic Acid (HOAC): 37.5 mL glacial acetic acid in 412.5 mL distilled water.

- B.) Ethyl Ether
- C.) Ammonium Hydroxide (NH₄OH).
- D.) Methanol

III. Equipment:

- A.) 250 mL glass beakers (4).
- B.) Mortar and pestle.
- C.) 7.0 cm filter paper.
- D.) 250 mL volumetric vacuum flask.
- E.) Glass separatory funnels with stoppers and stopcocks (2).
- F.) Glass stir rod.
- G.) pH paper.
- H.) Glass pipettes.
- I.) Hot sand bath.
- J.) 5 uL autosampler syringe (for manual injections)
- K.) GC/MS: HP 6890/5973 or HP 7890A/5975C series

IV. Procedure:

A.) Extraction Using Ethyl Ether:

1. Observe peyote buttons or shavings and compare to literature.
2. Dry peyote buttons at 100 degrees for ½ hour.
3. Weigh out 2.5 g of sample in a tared 250 mL beaker. Be sure to include all parts of the button.
4. Grind the sample into finer pieces with a mortar and pestle. Add back to the beaker.
5. Fill beaker with 3 N HOAC so solution is just above the sample and so sample is saturated. Let sit overnight in a dark place (ex. drawer).
6. Vacuum filter with 3 N HOAC, making sure to rinse the beaker very well.
7. In a hood, extract with ethyl ether by pouring the filtered sample into a glass separatory funnel, add ethyl ether (5:1 organic to aqueous), stopper and invert a several times. Make sure to vent the funnel to let excess gas inside the funnel escape.
8. Remove stopper and allow the layers to separate. The ethyl ether layer will be on the top.
9. Drain the bottom layer directly into another glass separatory funnel and discard the ethyl ether layer into appropriate waste container.
10. Add NH₄OH until solution is basic.
11. Extract again with ethyl ether (5:1 organic to aqueous). This time discard the bottom layer into waste container and drain the ethyl ether layer in to a beaker labeled with the sample number.
12. Either let the sample evaporate overnight in a dark place, or place on a hot sand bath until dry.

13. Rinse the beaker with Methanol, swirling to make sure the sides of the beaker get hit with Methanol.

B.) Chromatography by GC/MS:

1. Manually inject, in the following order, 1.5 uL of the mescaline standard, blank, and sample(s) directly from the beaker into the GC/MS.
2. GC/MS conditions:
Method: HYD.M
Oven:
Equilibration Time: 0.50 min.
Initial Temp: 135°C
Oven Max: 325°C.
Rate: 10°/min.
Final Temp: 280°C
Run Time: 30 min.
Inlet:
Mode: split (50:1)
Initial Temp: 250°C
Pressure: 31.65 psi
Gas Type: Helium
Column:
Capillary: HP-1MS 25m x 200um x 0.33um
Max. Temp: 300°C
Initial Flow: 1.0 mL/min.
3. If mescaline is present in sample, the instrument will detect a total ion peak at its retention time and will generate a report along with accompanying chromatograph and spectra. The spectra will contain the identity of the peak and its ion abundance (see graph, last page).

V. Results:

- A.) Record results of the GC/MS in logbook. Then transfer the results to appropriate evidence cards that came with the actual samples. Be sure to include date of analysis, results, the number of tests performed per sample, and initials.
- B.) All reports generated from the instruments should be filed so that they may be accessed at a later date, if necessary.